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DAMAGE DETECTION AND SELF-REPAIR IN HOLLOW GLASS FIBER FABRIC-REINFORCED EPOXY COMPOSITES VIA FIBER FILLING

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Abstract

Hollow glass fiber reinforced epoxy matrix composites were produced to study whether the damage development can be followed and the self-repair can triggered by filling the fibers with suitable additives. Composite plates were manufactured by the hand layup and vacuum assisted resin transfer molding techniques. To detect subcritical transverse impact damage, hollow fibers were filled with an ultraviolet fluorescent dye, whereas for self-repair, they were filled with a polyester resin along with the corresponding accelerator. The healing process was induced at different temperatures and continued for different durations. It was demonstrated that the targeted damage detection and self-repair can be achieved using thin (10-13 μm outer diameter) reinforcing hollow fibers. The self-repairing ability was demonstrated in three point bending tests and the healing was confirmed by inspection with scanning electron microscopy.

1. Introduction

Microcracks in fiber reinforced composites, which are initiated mostly under fatigue and impact conditions, should be repaired before fast crack propagation and catastrophic failure occur. The microscopic damage may be diminished by self-healing, provided that a type of repair liquid flows into the cracks and fills them properly. The latter means that the repair liquid adheres to the crack flanks and provides suitable cohesive strength at the same time. The idea of self-reparation comes from nature. As trees bring resin to their injuries, or as mammals heal their skin injuries by bleeding, the composites may also store some healing liquid. Recommendations for the storage of healing agents in composites have already been made [1]. One possible element for storing the liquid is the hollow fiber, embedded into the matrix, which can transport the healing liquid wherever it is necessary. The healing liquid flows into the crack and restores the connection between the broken parts [2]. Self-repair and damage assessment are of interest for concrete parts [3, 4], polymers [5, 6] and related composites, as well [7-10]. Hollow fibers may also contain different "indicator" liquids, which can help us to detect the damage development. Storing the healing agent in hollow fibers in composites is more advantageous than storing it in microcapsules [11-13], or in vascular networks [14-16], because they can store larger amounts than microcapsules and still provide efficient reinforcement.

Motuku *et al.* [17] used different solid and hollow tubes in composites. Their basic concept for self-repair was to use hollow fibers filled with suitable agents alongside solid reinforcing fibers. They examined the alteration of the energy absorbing capability of plates after impact-induced damage and after migration of the healing agent to the damaged areas. Vinylester matrix-based self-repairing composite (SRC) plates were their model materials. It was demonstrated by optical microscopy that the hollow fibers do not alter the failure mode compared to that of conventional composites. The SRCs with hollow fibers were damaged by low velocity impact in the same manner as conventional composites.

Trask *et al.* [18] used hollow glass fibers (HGF) for the storage of the repairing agent. The outer diameter of the fibers was 60±3 μm and the degree of hollowness was 55% (the ratio of the outer to the inner diameter). HGFs were embedded into carbon fiber-reinforced composites, and the hollow fibers were filled with a healing agent to study the self-repairing function. The matrix of the composite and the healing liquid were both two-component epoxy resins. Inside the laminate, the HGFs were placed 70 and 200 μm apart. The fracture of the specimens was executed with a steel ring pushed onto the specimens with 1700 and 2000 N loads. The bending strengths of the reference specimens (without HGF), undamaged SRC, the damaged and healed SRC were measured. The healing fibers placed at 70 μm deteriorated the bending strength of the undamaged laminates markedly (8%), but the results after healing were better because of the greater amount of healing material (1700 N: 91%, 2000N: 89% residual strength) than in the other sample with the fibers placed 200 µm apart (1700 N: 90%, 2000 N: 80%). Contrarily, the bending strength reduction caused by the hollow fibers was lower in the case of 200 µm placement (2%).

Pang and Bond [7] filled the hollow fibers with resin mixed with ultraviolet (UV) fluorescent dye so the self-repairing function could be triggered by an UV lamp. Thus, the position of the cracks and the healing process caused by the flow of the resin flow into the cracks could be monitored. Laminates were prepared with outer layers of 0/90° lay-up using conventional E-glass fabrics and with inner layers using borosilicate HGF unidirectional fabrics. The outer diameter of the HGFs was 60 µm and the hollowness was 50%. The 0° directional HGFs were filled with the resin component and the 90° directional HGFs were filled with the hardener mixed with a UV fluorescent dye. Specimens were fractured after different periods (0, 3, 6 and 9 weeks) to examine the effect of time on the healing ability of the curing agent. A 24 hour curing period was given to the composite for healing via resin crosslinking. Four point bending tests were performed on the specimens. The bending strength of the damaged, unfilled specimens was 25% lower than that of the undamaged, unfilled ones. The first self-repairing tests were performed immediately after filling the HGFs of the cured composite. The bending strength of these specimens was 93% of that of the unfilled, undamaged ones. With elapsed time the healing ability was reduced, and after a 9 week period it ceased. Obviously, the healing resin was affected by the storage conditions and the environment, and thus it could no longer fulfill its role as a healing agent.

Regarding the previous studies, it can be concluded that a self-healing function is possible with hollow fiber reinforcement, but the required diameter of the fibers is well above the ideal diameter that does not deteriorates the mechanical properties of the composites. The aim of our research was to develop a damage-detecting and a self-repairing composite which is reinforced with thin hollow fibers. Thin hollow fibers should ensure a reinforcing function. For the healing agent, a polyester resin was selected because it is less sensitive to the mixing ratio of the components than the epoxy resin used in the previous studies [7, 18-21]. A further aim of our study was to define an impact examination method that can be easily reproduced, which allows us to determine the healing ratio adequately. The effects of the repair periods and the repair environments on the healing rate were also examined.

2. Materials

The HGF fabric was obtained from R&G Faserverbundwerkstoffe GmbH (Waldenbuch, Germany). The fibers are made of H-glass, using a patented mixture of an alkali free aluminum-borosilicate by a proprietary manufacturing method. The nominal outer diameter of the fibers was 10-12 μm, while the inner diameter was 5-6 μm. The weight of the fabric is 160 g/m^2, and the structure was a 0/90° atlas weave.

For the matrices of the composites manufactured by vacuum assisted resin transfer molding (VARTM) and hand layup (HLU), respectively ipox MS90 (Ipox, Budapest, Hungary) and Ciba 5082-5083 (Ciba, Basel, Switzerland) resin systems were selected. Eporezit AH12 resin was chosen as epoxy carrier for the indicator liquid. It was "colored" by the UV fluorescent indicator, viz. Keystone Rhodamine B Base (Keystone, Chicago, USA). For damage detection in the composites, their matrices were painted with Eporezit SZPM white dye. This was incorporated at 5 wt% in order to ensure a better resolution for damage inspection. The coloring is important because without it, the painted indicator liquid can be observed through the glass fibers and the initially transparent matrix. "Coloring" of the matrix in this way is a straightforward method to support the detection of the indicator liquid, which is flowing onto the surface of the damaged composite. The healing agent was Polimal 1058 (Polimal, Poland) injectable polyester resin system.

3. Technologies

3.1. Manufacturing of the composite plates

During the manufacture of the HGF reinforced composites it was important to avoid matrix resin flow inside the fibers. In case of the HLU, the laminate was put under weights and placed on a glass plate to guarantee an equal distribution of the resin within the composite. Composite plates were cured for 24 hours at room temperature (23°), followed by an 80°C heat treatment in an oven for 8 hours.

3.2. Filling of the hollow glass fiber reinforced composites with liquids

It is very important when filling the hollow fiber reinforced plates that the ends of the fibers should not be clogged. After cutting the specimens, they were ultrasonically cleaned and were conditioned in a Heraeus UT6 (Hanau, Germany) air drying oven for 10 hours at 60°C. The filling layout is shown in Figure 1.

After drying, one edge of the specimens was placed into a vacuum bag (1) with a flow medium net (2) on both sides. Therefore, the hollow fibers remained accessible for the resin. The other end of the specimens was placed into the filling liquid (3). Recall that this was either the polyester healing resin or the colored ("painted") epoxy resin. To become self-repairing the hollow fiber reinforced composites were filled with a catalyzed unsaturated polyester resin and with its initiator (methyl ethyl ketone peroxide) dissolved in dimethyl phthalate (trade name: Butanox M50, Amersfoort, Netherlands, Akzo Nobel Company). To reach the adequate mixing ratio (resin/initiator, 100:5) only a smaller portion of the fibers had to be filled with the initiator (maximum 10%). For this purpose a special specimen was required. Therefore, to control the positions of the peroxide-containing fibers the composite plate was notched with a ribbon-saw. Where the composite was not notched, the fiber remained intact and could be filled with peroxide. On the side, where the filling with peroxide was executed, the composite plate was not notched (4). The preparation of the specimens begun with saw-cutting yielding the "notches", so unnotched sections remained (4). The width of each unnotched section was 3 mm and the distance between them was 22 mm. Prior to filling the fibers all edges of the plates were cleaned with an ultrasonic cleaner in acetone for 30 minutes. Thereafter they were dried for 12 hours at 60°C. The first step of the filling process was to fill the specimens with peroxide. The unnotched sections (4) were immersed into the liquid, and the other ends of the specimens were placed under suction by a vacuum bag. After filling the vertical longer fibers with peroxide, they were isolated by plasticine. In the second step, the polyester resin was introduced into the shorter fibers prepared by the saw cutting from the upper side of the specimens, so that gravity also supported the filling. When the filling was finished, the vacuum bags were removed from both sides of the specimens. In this way, all of the vertical fibers were filled - the longer ones with peroxide and the shorter ones with the healing polyester resin. Afterwards, the initial horizontal fibers in the fabric should be filled with the healing polyester resin to achieve the required resin/initiator ratio. Therefore, the specimens were rotated 90° and immersed into the healing polyester (3) according to configuration a) in Figure 1. Finally, a 6-7% initiator/resin ratio was achieved depending on the filling efficiency.

3.3. The method of specimen fracture and the measurement of the rate of self-repairing

Different types of fracture methods have been studied [7, 18, 22, 23], and utilizing those previous experiences, a method was developed that has repeatable fracturing and allows the measurement of residual strength. Specimens (width×length=23×60 mm) were cut from the unfilled and filled hollow glass fiber fabric reinforced composite plates. The specimens were damaged with a Ceast Fractovis (Italy) falling weight impact test machine. The energy of the fracture was set so that the residual strength of the specimens could be measured with three point bending. During impact-damaging, the whole surface of the specimen was put on a rubber sheet with a Shore A hardness 64±3. The pre-damaged specimens were subjected to three point bending tests, performed by a Zwick Z020 (Germany) device, to measure their residual strength values. The span length of the three point bending was 40 mm in accordance with the EN ISO 14125 [24] standard, and the test speed was 2 mm/min.

4. Filling of hollow fiber reinforced composites with the indicator liquid

Hollow fiber reinforced composites were manufactured, and they were filled with indicator liquid in order to examine the effects. The matrix of the first specimen was not colored. Four layers of hollow glass fabric were impregnated with Ciba LY 5082/HY 5083 resin system, and the thickness of the plate after curing was 1.2 mm. The 0/90°directional hollow glass fiber reinforced composites were filled with indicator liquid in only one direction in the first case (only the 0° directions) and both directions in the second case (in both the 0 and 90° directions). The specimens were hit at four and five different places. All of the sets were loaded with 3.62 kg. The height of the falling weight was set at 0.02, 0.04, 0.06, 0.08 and 0.1 m, and the corresponding energies were 0.71, 1.42, 2.13, 2.84 and 3.55 J. It was obvious that the identification of the damage is difficult without “painting” of the composite matrix because the indicator liquid is visible through the glass fibers and the matrix. Therefor, the indicator liquid in the cracks cannot be distinguished from the indicator liquid in the fibers under the UV lamp. It can be observed, however, that it is sufficient to fill the fibers only in one direction with the indicator liquid because it penetrates into the cracks accordingly (Figure 2).

On the impacted side of the composites the damage areas, produced by the hits, are poorly resolved. Filling the specimens with the indicator liquid from both directions did not help to identify the impacted spots. For this reason, no pictures are included. On the side opposite the impact, the contrast between the cracks and the surrounding is stronger, so the cracks are easily perceptible even for the naked eye. On the side opposite of the impact, even the crack due to the hit with the smallest energy (0.71 J) is clearly visible. The reason for this phenomenon is the growing area of the delamination in the thickness direction from the point of the hit. In the delamination area, the fibers break [25], and thus more indicator liquid can flow into the wider crack on the side opposite the impact. The cracks in the colored matrix composite could be better observed, even when only 0° directional fibers were filled (Figure 3).

The coloring of the matrix clearly helped us to identify the cracks. Except for the smallest energy impact, all of the cracks at higher incident energies can also be clearly observed from the impact side. On the side opposite the impact the cracks are also very well resolved by the naked eye, but they are more easily identified under UV irradiation. On the side opposite the impact, both the 0 and the 90° directional fibers are broken, and the liquid is released into the whole crack even when the indicator filling was from one direction, but with the matrix colored. This finding suggests that self-repairing could work if one part of the fibers was filled with one component of the healing agent, whereas the other part of the fibers was filled with the hardener of the healing agent.

5. The examination of hollow fiber reinforced composites filled with polyester resin and peroxide

5.1. Specimens prepared with HLU

HGF reinforced composites were prepared by the HLU technique as described above. They were filled with the components of a polyester resin system, and the self-repairing ability was examined. The nominal size of the specimens was 24×1.25×60 mm. The energy of the impact was adjusted so that after the impact the residual strength and modulus could be measured in three point bending. The bending strength and the bending modulus were compared on different types of specimens: unfilled – undamaged, unfilled – damaged, filled – undamaged, filled – damaged, filled – healed. During the preliminary fracture tests it was observed that the filled specimens can absorb more energy than the unfilled ones. The energy of the incident impact was selected so that the residual strength remained well measurable. The weight of the falling dart was 3.62 kg, the falling height of the unfilled specimens was 40 mm (1.42 J), whereas the falling of the filled specimens was 110 mm (3.91 J). The filling of the hollow fibers increased the energy absorbing property of the composite specimens; the low impact energy did not result in fracture in the specimen, so the impact energy had to be increased so the healing could be measured. The formation of the impact damage on a specimen is seen in Figure 4.

The three point bending analysis was executed immediately after the impact damage. The related specimens are referred to as the filled – damaged ones. Another series of these specimens were put into an oven at 60°C for 12 hours, before determining the residual flexural properties. The results of the measurements are displayed in Figures 5. and 6.

The results show that the filling has a positive effect on the resistance to a bending load. The bending strength of the filled – undamaged specimens is higher by 14 % than that of the unfilled – undamaged one, whereas there is no significant difference in their moduli. The filling also improved the impact resistance: whereas the impact energy was 175% higher in case of the filled specimens, the bending strength and the modulus were 103% and 28%, respectively, higher than those of the unfilled specimens. The better results of the filled specimens are due to i) the incompressibility of the liquids inside the hollow fibers, and ii) the absorption of energy because of the friction between the liquid polyester resin and the fiber inner wall. If all of the fibers (with an outer diameter of 13.1 μm, and an inner diameter of 8.1 μm) of a composite with 40 V/V% hollow fiber content were filled, the weight of the filled specimens would be 13% higher than the unfilled ones, but they would still be 15% lighter than the composites reinforced with solid fibers. The healing has been proved unequivocally. Comparing the results of the filled – healed specimens to the filled – damaged specimens, the bending strength and modulus are higher by 23% and 20%, respectively.

5.2. Specimens manufactured by VARTM

To enable the comparison of the results, a HGF reinforced epoxy matrix composite filled with polyester resin and with peroxide was manufactured by another manufacturing method, as well. For that purpose, the VARTM method was chosen, which yields much more homogenous material properties than HLU. Further, instead of 3 layers, as are used in HLU, 5 layers of 160 g/m^2 hollow glass fabric were applied to achieve a thickness similar to that of the HLU series. The nominal size of the specimens was 24×1.2×60 mm. Despite the two extra layers, the thickness is lower because of the higher fiber content achieved by VARTM. Because of the higher fiber content and the resultant higher mechanical properties, a higher impact energy was chosen to damage the specimens. The weight of the dart was set at 8.62 kg, and the height was 110 mm. This resulted in an incident impact energy of 9.3 J, which was applied for both unfilled and filled specimens. The test method was the same as in case of the HLU specimens, i.e., 5 specimens per group were measured. The healed specimens were placed into a 60°C oven for 12 hours, and then the three point bending tests were executed. The results of the examinations are seen in Figures 7. and 8.

The results obtained on the VARTM-manufactured specimens show a tendency similar to those of the HLU-manufactured specimens. The filling improved the bending properties. There is no significant difference between bending strength of the unfilled – undamaged and the filled – undamaged specimens, however, the bending modulus was increased by the filling by 16%. Filling also increased the residual flexural properties: the bending strength was 178% higher and bending modulus was 11% higher for the filled specimens. The improved properties of the filled specimens also resulted from the energy absorbing ability of the liquid inside the hollow fibers, which increases both the strength and the modulus. The bending strength of the healed specimens was 25% higher, and the bending modulus was 46% higher compared to the results obtained on the filled – damaged specimens.

5.3. Long term healing at room temperature

The self-repair was also tested without putting the specimens into the oven. The related specimens were also manufactured by the VARTM method using 5 plies of hollow glass fabric. The previous test protocol as adapted, however, with one difference: After the damage the specimens were not heat-treated, but were instead stored at 23°C for 120 hours prior to performing the bending tests. The results of the flexural measurements can be seen in Figures 9. and 10.

The bending strength of the healed specimens was 20% higher, and the bending modulus was 26% higher compared to the filled – damaged specimens. It is observed that when sufficient curing time for the healing agent is provided, the self-repairing process can proceed.

5.4. Scanning electron microscopy

Healed specimens of the hollow fiber reinforced composites were embedded into resin, and it was polished to examine the traces of self-repair with a JEOL JSM 6380LA scanning electron microscope (Japan). An electron micrograph can be seen in Figure 11.

In the SEM picture in Figure 11, the locations of the fibers before they were separated by the crack are well visible – cf. arrows. Under the effect of the impact load delaminations with fiber fractures occurred. The healing agent flowed out from the fibers and filled the cracks. The cross-linked healing agent is observed inside the crack. Thus, the integrity of the structure was restored, and because of this, the bending properties improved after the healing phase compared to the damaged state.

6. Summary

Hollow fiber reinforced composite plates were produced and the fibers were filled either with an indicator liquid or with a polyester resin to visualize subcritical damage or to investigate the self-repairing function, respectively. In this work, thin hollow glass fibers (13 μm outer diameter) were applied to ensure major reinforcement in contrast to work in the literature (30 μm and larger outer diameters). Fluorescent dye proved to be an efficient tool for damage detection under UV illumination. The bending properties and the impact resistance were improved by the filling of the hollow fibers. The specimens, manufactured by the HLU and VARTM methods, were damaged in a falling weight impact testing machine, and they were left to heal at 60°C and 23°C for 12 and 120 hours, respectively. It was demonstrated that the self-repair can be triggered by filling the hollow fibers with a polyester resin system (catalyzed resin and initiator in a given ratio). As a healing method in self-repairing materials, the curing of the polyester resin is more secure than the curing of epoxy resin used in the prior literature. It is more secure because the curing is not so sensitive to the mixing ratio and method. The curing of the polyester resulted in at least a 20% improvement in the bending properties of the pre-damaged specimens after healing compared to the reference specimens without healing. With polyester resin a larger improvement could be ensured for a variety of conditions than were obtained with epoxy resin in the literature. Additionally, the healing can be achieved at both ambient and higher temperatures provided that the holding time is selected accordingly. Further investigation is needed to test whether the polyester healing agent also works when the related composites are stored for a longer time prior to being damaged.

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Caption of figures

Figure 1. Vacuum-assisted filling of the hollow fiber reinforced specimens with one a component (a) and a two component healing agent (b). 1 – vacuum bag, 2 – medium net, 3 – filling liquid, 4 – unnotched section

Figure 2. Specimens filled from one direction under a UV-C lamp from the direction of impact (a) and from the side opposite the impact (b)

Figure 3. Colored matrix of a specimen filled from one direction under a UV-C lamp from the side of impact (a) and from the opposite side of the impact (b)

Figure 4. The formation of the impact damage

Figure 5. The flexural strength of the specimens manufactured by HLU

Figure 6. The flexural modulus of the specimens manufactured by HLU

Figure 7. The flexural strength of the VARTM specimens

Figure 8. The flexural modulus of the VARTM specimens

Figure 9. The flexural strength of the specimens healed at room temperature

Figure 10. The flexural modulus of the specimens healed at room temperature

Figure 11. Scanning electron micrograph of the healing agent that has flowed into the crack

Figure 1

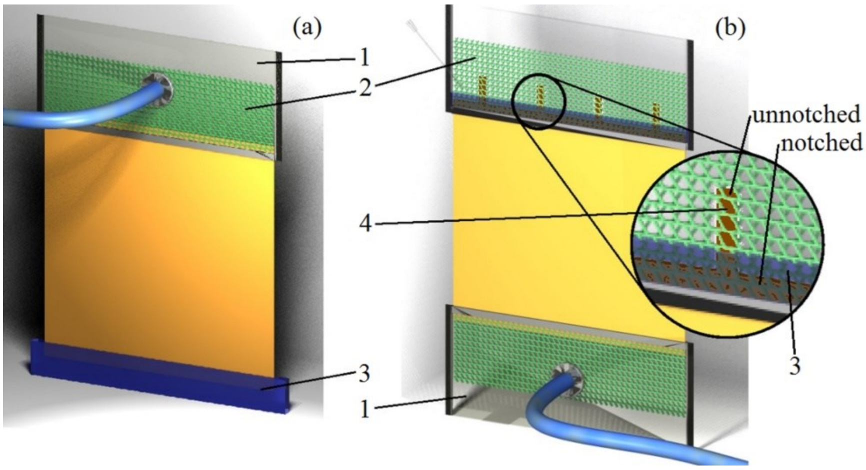

Figure 2

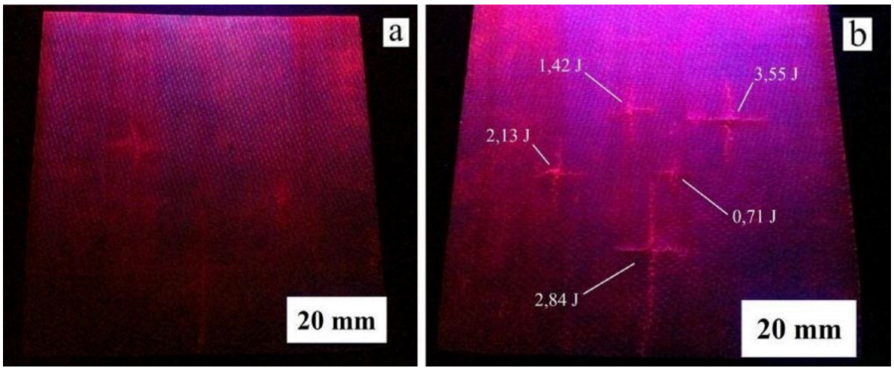

Figure 3

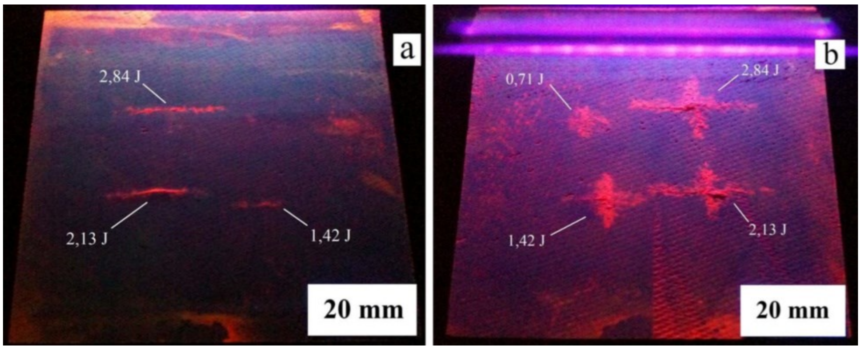

Figure 4

Figure 5

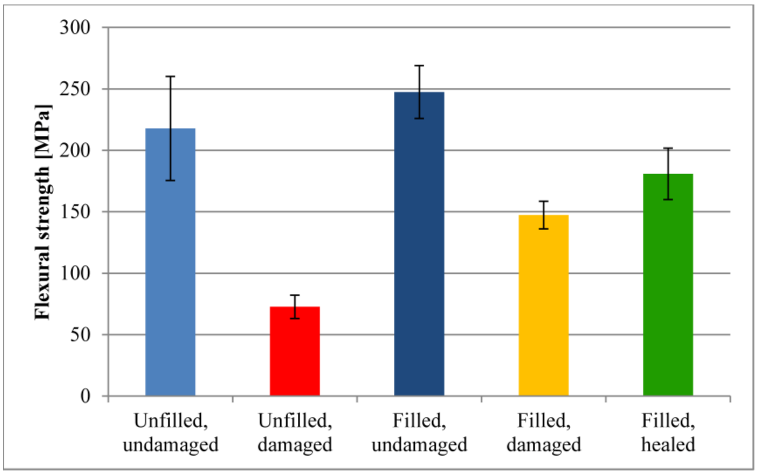

Figure 6

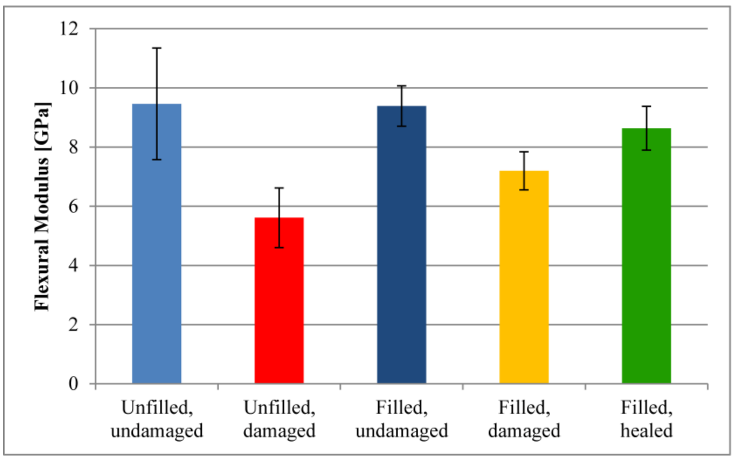

Figure 7

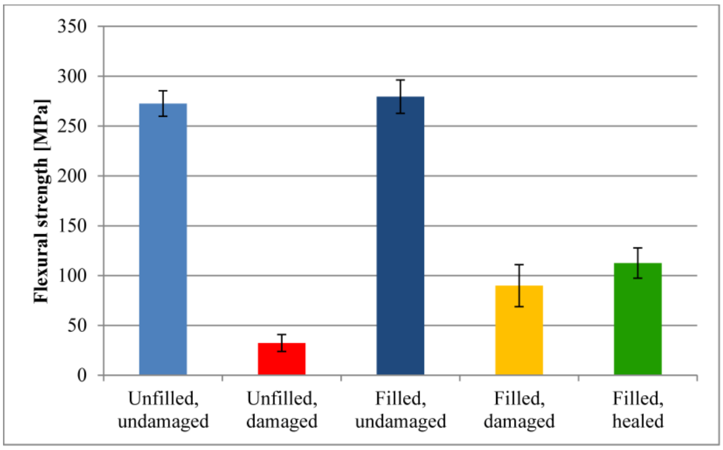

Figure 8

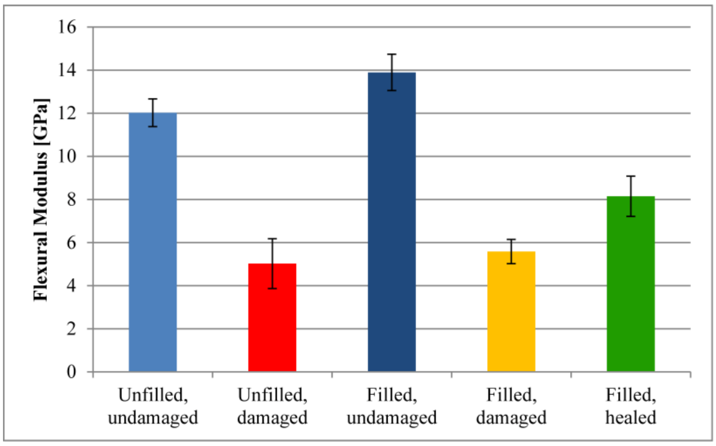

Figure 9

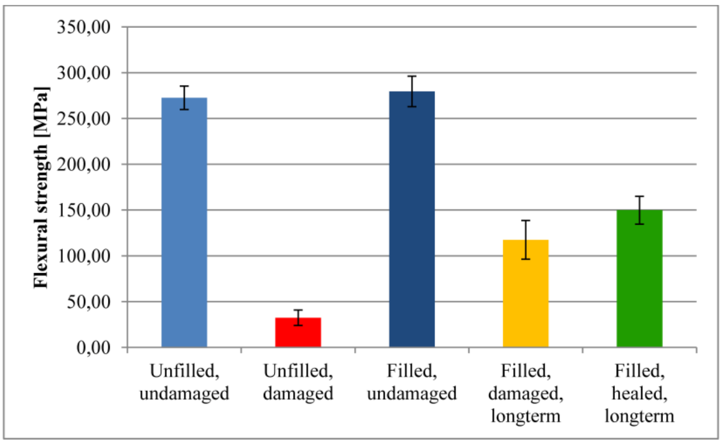

Figure 10

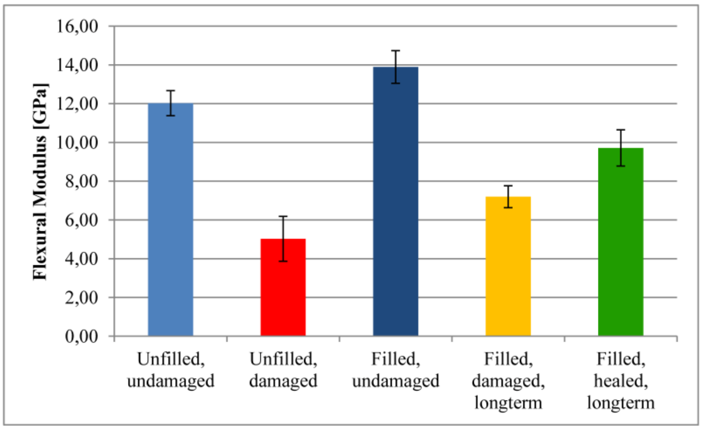

Figure 11